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SPECIFICATION FOR GRAPHITE, FLAKE, FOR LUBRICANTS

(First Revision)

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BUREAU OF INDIAN STANDARDS
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Indian Standard
SPECIFICATION FOR GRAPHITE, FLAKE, FOR LUBRICANTS
(First Revision)

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Indian Standard

SPECIFICATION FOR GRAPHITE, FLAKE, FOR LUBRICANTS

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 14 July 1967, after the draft finalized by the Lubricants and Related Products Sectional Committee had been approved by the Chemical Division Council and the Mechanical Engineering Division Council.

0.2 This standard was first published in 1954. Amendment No. 1 to this standard was issued in April 1962 and Amendment No. 2 in October 1962. As a result of comments subsequently received, changes have been made in this revision in methods of test for loss on heating, ash and non-graphitic carbon and in the method of drawing samples and criteria for conformity. Also a requirement for freedom from abrasive matter has been included.

0.3 In the preparation of this standard, assistance has been derived from Indian Defence Service specification IM 1690(b) 'Graphite; flake'; issued by the Ministry of Defence; and Material specification No. DTD 806 : 1950 'Graphited grease' issued by the Ministry of Aviation, UK.

0.4 For determining fineness, the aperture sizes of sieves are based on IS : 460-1962*. Where these sieves are not available, other equivalent standard sieves, as judged by the aperture may be used.

0.5 This standard contains clauses **3.1** and **B-3.2** which call for agreement between the purchaser and the supplier.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960†. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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*Specification for test sieves (*revised*).

†Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for graphite, flake, intended chiefly for use in the manufacture of lubricants.

2. REQUIREMENTS

2.1 Description — The material shall be genuine good quality graphite. It shall be dry, steel grey in colour, having a metallic lustre, and a smooth and soapy feel. It shall be free from grit, grease and visible impurities.

2.2 Fineness — The material shall wholly pass through 200-micron IS Sieve (*see* IS : 460-1962*); and not more than 5·0 percent (by weight) of the material shall be retained on 125-micron IS Sieve (*see* IS : 460-1962*).

2.3 The material shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in Appendix A.

TABLE 1 REQUIREMENTS FOR GRAPHITE, FLAKE FOR LUBRICANTS

Sl No. (1)	Characteristic (2)	Requirement (3)	Method of Test (4)
i)	Loss on heating, percent by weight, *Max*	1·25	A-2
ii)	Petroleum ether-soluble matter, percent by weight, *Max*	0·50	A-3
iii)	Water-soluble matter, percent by weight, *Max*	1·25	A-4
iv)	Acidity of water extract	To satisfy the requirement of the test	A-5
v)	Ash, percent by weight, *Max*	5·0	A-6
vi)	Non-graphitic carbon, percent by weight, *Max*	3·0	A-7
vii)	Freedom for abrasive matter	To pass the test	A-8

3. PACKING AND MARKING

3.1 Packing — The material shall be packed as agreed to between the purchaser and the supplier.

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*Specification for test sieves (*revised*).

3.2 Marking — The containers shall be marked with the name of the material and the net weight in the container, name of the manufacturer and recognized trade-mark, if any, and batch number or code number.

3.2.1 The container may also be marked with the Standard Mark

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in Appendix B.

APPENDIX A

(*Clause* 2.3 and *Table* 1)

METHODS OF TEST FOR GRAPHITE, FLAKE, FOR LUBRICANTS

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF LOSS ON HEATING

A-2.1 Procedure — Weigh accurately about 5 g of the material in a tared squat-type weighing bottle. Keep it in an air-oven maintained at 105 ± 1°C for 2 hours. Cool the weighing bottle in a desiccator and weigh.

A-2.2 Calculation

$$\text{Loss on heating, percent by weight} = 100\frac{w}{W}$$

---

*Specification for water, distilled quality (*revised*).

where

w = loss in weight in grams on heating, and

W = weight in grams of the material taken for the test.

A-3. DETERMINATION OF PETROLEUM ETHER SOLUBLE MATTER

A-3.1 Reagent — Petroleum ether conforming to grade 60/80 of IS : 1745-1966*.

A-3.2 Procedure — Weigh accurately about 10 g of the material and extract with petroleum ether in a Soxhlet apparatus for 2 hours. Filter the petroleum ether extract through a filter paper into a tared flask. Distil off the solvent and remove the last trace of it in vacuum. Cool the flask and weigh to constant weight.

A-3.3 Calculation

$$\text{Petroleum ether-soluble matter, percent by weight} = 100\,\frac{w}{W}$$

where

w = increase in weight in grams of the flask, and

W = weight in grams of the material taken for the test.

A-4. DETERMINATION OF WATER SOLUBLE MATTER

A-4.1 Procedure — Weigh accurately about 5 g of the material and boil with 200 ml of water for 5 minutes. Make up the volume to 250 ml with water. Filter the solution. Reject the first 50 ml of the filtrate and take an aliquot portion (say V ml) from the remaining solution and evaporate to dryness on a water-bath. Dry the residue to constant weight in an air-oven maintained at 105 ± 1°C. Preserve the remaining solution for test in **A-5**.

A-4.2 Calculation

$$\text{Water-soluble matter, percent by weight} = \frac{25\,000\,X}{VW}$$

where

X = weight in grams of the residue obtained,

V = volume in millilitres of the solution taken for the test, and

W = weight in grams of the material taken for the test.

---

*Specification for petroleum hydrocarbon solvents (*first revision*).

A-5. TEST FOR ACIDITY OF WATER EXTRACT

A-5.1 Reagents

A-5.1.1 *Sodium Hydroxide Solution* — 0·1 N.

A-5.1.2 *Phenolphthalein Indicator* — Dissolve 0·1 g of phenolphthalein in 60 ml of rectified spirit (conforming to IS : 323-1959*) and dilute with water to 100 ml.

A-5.2 Procedure — To 50 ml of the solution as prepared under **A-4.1** add 1 drop of sodium hydroxide solution and 1 drop of phenolphthalein indicator solution and mix.

A-5.2.1 The material shall be taken to have satisfied the requirement of the test if the test solution is pink or red in colour.

A-6. DETERMINATION OF ASH

A-6.1 Procedure — Weigh accurately about 2 g of the material in a tared platinum dish and incinerate till the residue in the crucible is constant in weight. Cool in a desiccator and weigh.

A-6.2 Calculation

$$\text{Ash, percent by weight} = 100\frac{w}{W}$$

where

w = weight in grams of the residue, and
W = weight in grams of the material taken for the test.

A-7. DETERMINATION OF NON-GRAPHITIC CARBON

A-7.1 Apparatus

A-7.1.1 *Muffle Furnace* — having a thermoregulator and maintained at 440 ± 5°C. Temperature of the furnace shall be noted by introducing a suitable thermometer in the centre of the furnace.

A-7.2 Procedure — Weigh accurately about 2 g of the material into a silica dish fitted with cover and note the weight of the dish with the material. Place the dish in the centre of the furnace in such a manner that 40 percent of the centre area of the muffle is occupied by the dish. Heat the sample for 16 hours in the furnace, then cool it and its contents in a desiccator and weigh.

NOTE — The heating for 16 hours should preferably be done at a stretch. If this is not practicable, it should be done in two equal instalments of 8 hours each on two successive working days.

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*Specification for rectified spirit (*revised*).

A-7.3 Calculation

$$\text{Non-graphite carbon, percent by weight} = 100\frac{(W_1 - W_2)}{W} - (A + B)$$

where

W_1 = first weight in grams of the dish and contents,
W_2 = second weight in grams of the dish and contents,
W = weight in grams of the sample taken for the test,
A = percent loss on heating (*see* **A-2**), and
B = percent petroleum ether-soluble matter (*see* **A-3**).

A-8. TEST FOR FREEDOM FROM ABRASIVE MATTER

A-8.1 Procedure — Take a pair of flat circular brass plates with chamfered edges and of approximately 75 mm diameter, polished successively with fine emery cloth and finally with rouge to give a mirror surface, Weigh about 2 g of the material, squeeze it between the polished faces of the brass plates and rub then vigorously for 1 minute. Wash the plates with a suitable solvent and examine for visible scratching.

A-8.1.1 The material shall be taken to have satisfied the requirements of the test, if there are no visible scratches on any of the two plates.

APPENDIX B

(*Clause* 4.1)

SAMPLING OF GRAPHITE, FLAKE

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall not be taken in an exposed place.

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in suitable clean, dry and air-tight metal or glass containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.7 Each sample container shall be sealed air-tight by suitable means after filling and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

B-2. SCALE OF SAMPLING

B-2.1 All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute the **lot**. If a consignment is declared or known to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

B-2.1.1 Samples shall be tested from each lot for ascertaining conformity of the material to the requirements of the specification.

B-2.2 The number of containers (n) to be chosen from the lot shall depend on the size of the lot (N) as given below:

Lot Size N	*Sample Size* n
3 to 50	3
51 „ 200	4
201 „ 400	5
401 „ 650	6
651 „ 1 000	7

B-2.3 The containers to be selected for sampling shall be chosen at random from the lot and for this purpose, random number tables shall be used; in case such tables are not available, the following procedure may be adopted:

> Starting from any container, count them 1, 2, 3,..., etc, up to r and so on in a systematic manner, where r is the integral part of N/n. Every rth container thus counted shall be withdrawn from the lot to constitute the sample.

B-3. TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Preparation of Test Samples

B-3.1.1 Draw, with an appropriate sampling instrument, small portions of the material from different parts of each container selected for sampling and freshly opened. The total quantity of the material drawn from each container shall be sufficient to conduct tests for the characteristics given in **2** and shall be not less than 300 g.

B-3.1.2 Thoroughly mix all portions of the material drawn from the same container. Out of these portions, equal quantity shall be taken from each selected container and shall be well-mixed up together so as to form a composite sample weighing not less than 0·5 kg. This composite sample shall be divided into 3 equal parts, the first for the purchaser, the second for the supplier and the third for the referee.

B-3.1.3 The remaining portion of the material from each container (after the quantity needed for the formation of composite sample has been taken) shall be divided into 3 equal parts, each part weighing not less than 40 g. These parts shall be immediately transferred to thoroughly-dried bottles which are then sealed air-tight with stoppers and labelled with all the particulars of sampling given under **B-1.7.** The material in each such sealed bottle shall constitute an individual test sample. These individual samples shall be separated into 3 identical sets of samples in such a way that each set has an individual test sample representing each container selected. The first of these 3 sets shall be sent to the purchaser, the second to the supplier and the third shall be used as referee sample.

B-3.2 Referee Sample — The referee sample shall consist of the composite sample (**B-3.1.2**) and a set of individual samples (**B-3.1.3**) marked for this purpose and shall bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

B-4. NUMBER OF TESTS

B-4.1 Tests for loss on heating, petroleum ether soluble matter and for non-graphitic carbon shall be done on each of the individual samples.

B-4.2 Tests for all the other characteristics given under **2** shall be done on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 For Individual Sample — The mean and range for the test results for each of the characteristics tested on the individual samples shall be calculated as follows:

Mean x = sum of the test results divided by the number of test results, and

Range R = difference between the maximum and the minimum values of the test results.

B-5.1.1 The lot shall be declared to have satisfied the requirements for these characteristics if for each, the value of the expression ($x \pm 0·4\,R$) does not exceed the specified maximum limit.

B-5.2 For Composite Sample — The composite sample shall meet all the requirements tested on it.

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